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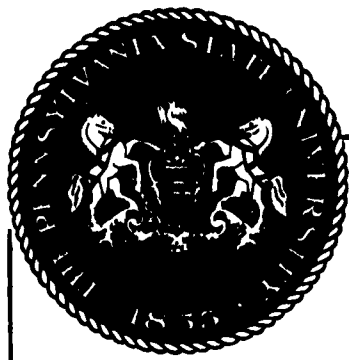

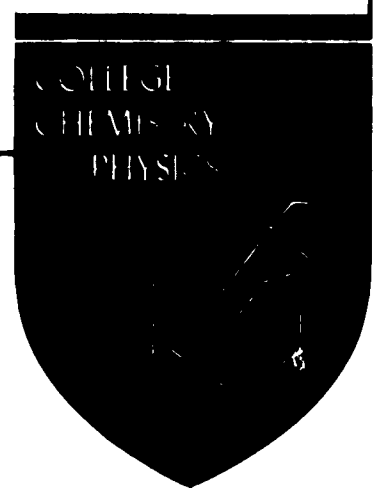

THE PENNSYLVANIA STATE UNIVERSITY

UNIVERSITY PARK, PENNSYLVANIA

Technical Report

A VERSATILE FIELD ION MICROSCOPE

S. B. McLane, Jr. and E. W. Müller

The Pennsylvania State University
University Park, Pennsylvania

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Principal Investigator: E. W. Müller

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ABSTRACT

An all metal design of the field ion microscope has proven to be both feasible and versatile. Adaptability, variable magnification, ruggedness, greater safety when used with liquid hydrogen cooling, and high voltage shielding characterize this model. A 6 inch diameter vertical screen, which is easily replaceable, facilitates its use. Application of this model to the study of different screen materials, the use of solid nitrogen cooling, and the study of grain boundaries is described.

TABLE OF CONTENTS

TABLE OF FIGURES

A VERSATILE FIELD ION MICROSCOPE

by

S. B. McLane, Jr. and E. W. Müller

I. INTRODUCTION

Originally field ion microscopes were constructed of Pyrex glass in sealed, baked systems similar to the field electron microscopes. The glassblowing and baking required each time the tip was changed occupied a period of several hours before operation of the microscope could be resumed. Later it was shown that when helium was used as the positive ion source that vacuum requirements for field ion microscopes were much less stringent than that required for field electron microscopes. This is due to the fact that helium has the highest ionization energy of all the elements (V_I = 24.47 ev) and all other substances which might contaminate the tip surface are ionized before they can approach the tip.[1] With this knowledge, we have for some time operated systems containing a number of grease sealed ground joints and still maintained clean surfaces over extended periods as can be demonstrated by successive photographs compared by light of complimentary colors which quickly show the presence or absence of even one single atom. This innovation greatly reduced the time required to change tips and to change screens, but the basic design of the system was still limited by the fragile nature of the glass

involved and the geometries still had to take into account the problems and techniques of glass blowing.

II. A FIELD ION MICROSCOPE CONSTRUCTED OF METAL

There was a need for a versatile field ion microscope which would be rugged and adaptable to a wide variety of experiments. To accomplish this, a system was designed utilizing a metal body for the microscope, a two inch aircooled metal oil diffusion pump (Veeco EP 2 AB) and metal bellows valves.

The microscope body provided two double O-ring flanges with a 5-3/4 inch opening, which could be used for screen mounting, and two openings for the microscope head which were so spaced as to give four different values for the tip-to-screen distance. Three other ports were provided on the sides to accommodate such things as gauges, getter assemblies or radiation sources. With the large double O-ring openings the only part of the whole system which had to be made of glass was a simple 8-1/2 inch diameter by 3/8 inch thick disk of Pyrex glass for the screen. The flat structure of the glass screen facilitated the application of the tin-oxide conductive coating, and a simple mask contained the fluorescent screen material to the desired area. Figure 1 shows the first arrangement using a glass microscope head.

With this arrangement it was difficult to get reproducible readings from the ionization gauge since the gauge

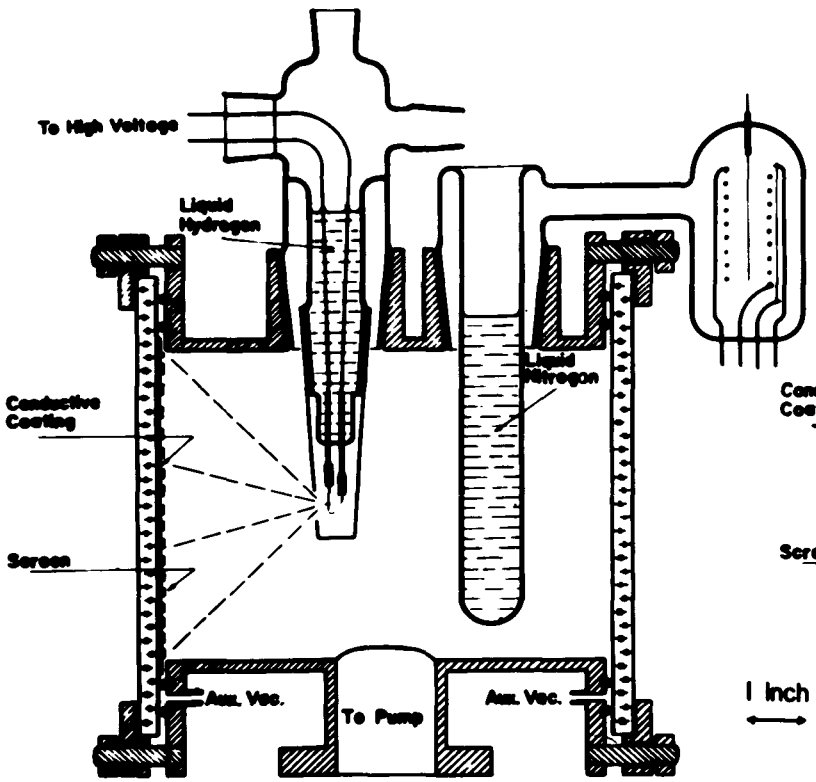

Figure 1 FIM Metal Body, Glass Head

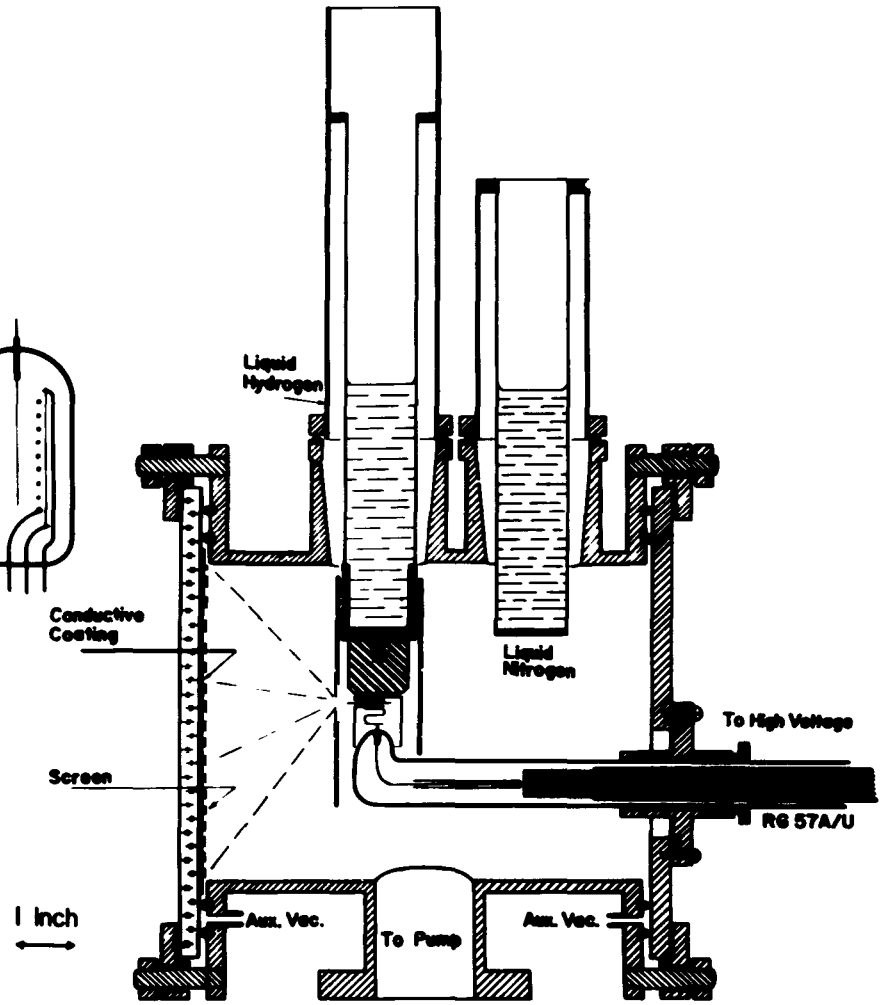

Figure 2 FIM Metal Body, Metal Head

1 inch

looked at a glass surface of the cold finger which had a temperature varying with the level of the liquid nitrogen. The commercial ionization gauge used (Veeco) had a molybdenum grid. On degassing, this sometimes vaporized to the walls and partly oxidized. The resulting molybdenum-oxide served as a virtual leak. As the gauge was both a source of gas and a pump for gas and was very sensitive both to the level of the liquid nitrogen in the cold finger and the temperature surrounding the ionization gauge, the readings for a steady pumping rate could vary by more than an order of magnitude. Vigorous and prolonged baking of the ionization gauge did not correct the situation. To solve this problem we built our own ionization gauge using only tungsten for all of the electrodes, and mounting the gauge inside the main body of the microscope, without any glass envelope, to a brass disk which replaced the rear glass disk in Figure 1. The tungsten electrodes were made with the same physical dimensions as the commercial gauge and it was found that the commercial gauge controller and meter not only controlled the gauge satisfactorily but that the calibration was the same as the commercial ionization gauge. This made ionization gauge came to an equilibrium reading faster and with less fluctuation than the commercial gauge and it degassed more rapidly and thoroughly. As it was located in the main microscope volume it gave a more direct value of the vacuum at the tip.

In the early history of this particular system, water or nitrogen etch[2] were encountered frequently, when the tip was cooled to 77°K. To overcome this a titanium getter was first used, but more satisfactory results were obtained with an activated charcoal getter. At first the charcoal was placed in a glass side arm heated by the blast of a hot air gun. This required ten minutes of heating and raised the ambient temperature of the narrow dark room. This arrangement was revised by placing a low voltage heater coil of nichrome ribbon internal to the charcoal. This reduced the time for equivalent degassing to three minutes. Care must be taken during the heating of the charcoal that the gases are pumped away without coming in contact with coldtraps at temperatures below that of solid CO_2, as the copious quantities of the CO_2 given off by the charcoal will condense there and are difficult to remove by further pumping. If in the present system the main cold trap for the diffusion pump has liquid nitrogen in it, it is satisfacotry to first heat the charcoal while pumping only with the forepump, and then to pump with the liquid nitrogen trapped diffusion pump after the heat has been removed, and the CO_2 development has stopped. The effectiveness of the activated charcoal getter has been illustrated by the fact that this field ion microscope has been operated continuously for more than 26 hours with liquid nitrogen cooling and no noticeable corrosion of the specimen by nitrogen or water vapor etch. Also the system has been

kept closed down without pumping for more than a week with a total pressure rise of less than one micron. The original charge of 50 cubic centimeters of 6-14 mesh activated cocoanut charcoal is still satisfactory after being in use for over one year.

Other major modifications of the system were made to improve the cooling of the cold mantle and thereby the helium supply to the tip, and to eliminate the problem of high voltage breakdown in the cooling liquids and gasses. First attempts at shielding the high voltage leads to a point below the minimum liquid level were not satisfactory as the shielding or insulating reduced the volume of the cooling liquid and transferred too much heat to it necessitating refilling with liquid hydrogen every six minutes. The problem of high voltage breakdown in the liquid hydrogen or liquid nitrogen and the cooling of the cold mantle were both solved by constructing a new microscope head of stainless steel which provided a metal to metal contact between the cold mantle and the container holding the cooling liquid, and by removing the high voltage leads from the cooling liquid. The first trial in this direction utilized two alumina insulators supporting the tip loop from above and below. The cold mantle provided a cooling path to the lower insulator. The two electrical connections to the tip loop were made by means of two coaxial helices of tungsten within the lower insulator. Due to space limitations it was tedius to make

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this connection when the microscope head was removed for changing tips and the extra length due to the second insulator gave difficulties in preventing high voltage breakdown to the body of the microscope. The present design is shown in Figure 2. In it a single Lavite insulator serves to cool the tip loop and to isolate it electrically. Lavite was used because it could be machined and then fired to hardness. This allowed the ceramic to be machined to size and to be tapped so that the insulator could be screwed to the cooling container and the two tip connectors screwed to the other end, as shown in Figure 5. The thermal conduction properties of the Lavite are inferior for this application to those of alumina or beryllia. Several experiments were conducted in an effort to find a satisfactory means of fastening metal parts to ceramics over a wide temperature range. The surface of a ceramic was coated with platinum by means of platinum bright, then electro-plated with copper and the parts soldered to the copper. So far this technique has not been mastered consistently. The arrangement in Figure 2 does accomplish excellent cooling of the cold mantle, and provides for convenient removal of the microscope head as well as alternate positions for the microscope head relative to the screen. Improvement in thermal conduction to the tip is still desired. Recently available data show that beryllium oxide at room temperature has a thermal conduction two orders of magnitude higher than

lava or magnesium silicate now in use.[3,4] Data is being sought on thermal values at lower temperatures and mounting methods for BeO. With this arrangement the high voltage is completely shielded from any accidental contact by the operator, and any high voltage breakdowns are confined within the cold mantle. It can also be assumed that the metal Dewar is much safer when operated with liquid hydrogen than a glass cold finger. There is always the possibility that a glass container might crack under the thermal stress, and the liquid hydrogen might be spilled and ignited by the applied high voltage. An incidental advantage accrued from constructing the cold finger and microscope head from stainless steel is that they turned out to be more efficient Dewars. The cold finger when filled with liquid nitrogen will last 5 hours and the microscope head will last 3 hours when filled with liquid nitrogen. The practice is to refill the microscope head every 27 minutes when working with liquid hydrogen. A photograph of the system in its current state is shown in Figure 3. The cabinet to the left contains the power supply, electrostatic voltmeter, ionization and thermocouple gauge controllers, photomultiplier microphotometer, and timers for photographic exposure and the refilling of liquid hydrogen.

In spite of the six Viton O-ring joints and two grease joints, vacuums suitable for the operation of the field ion microscope can be obtained readily. Ultimate vacuum with

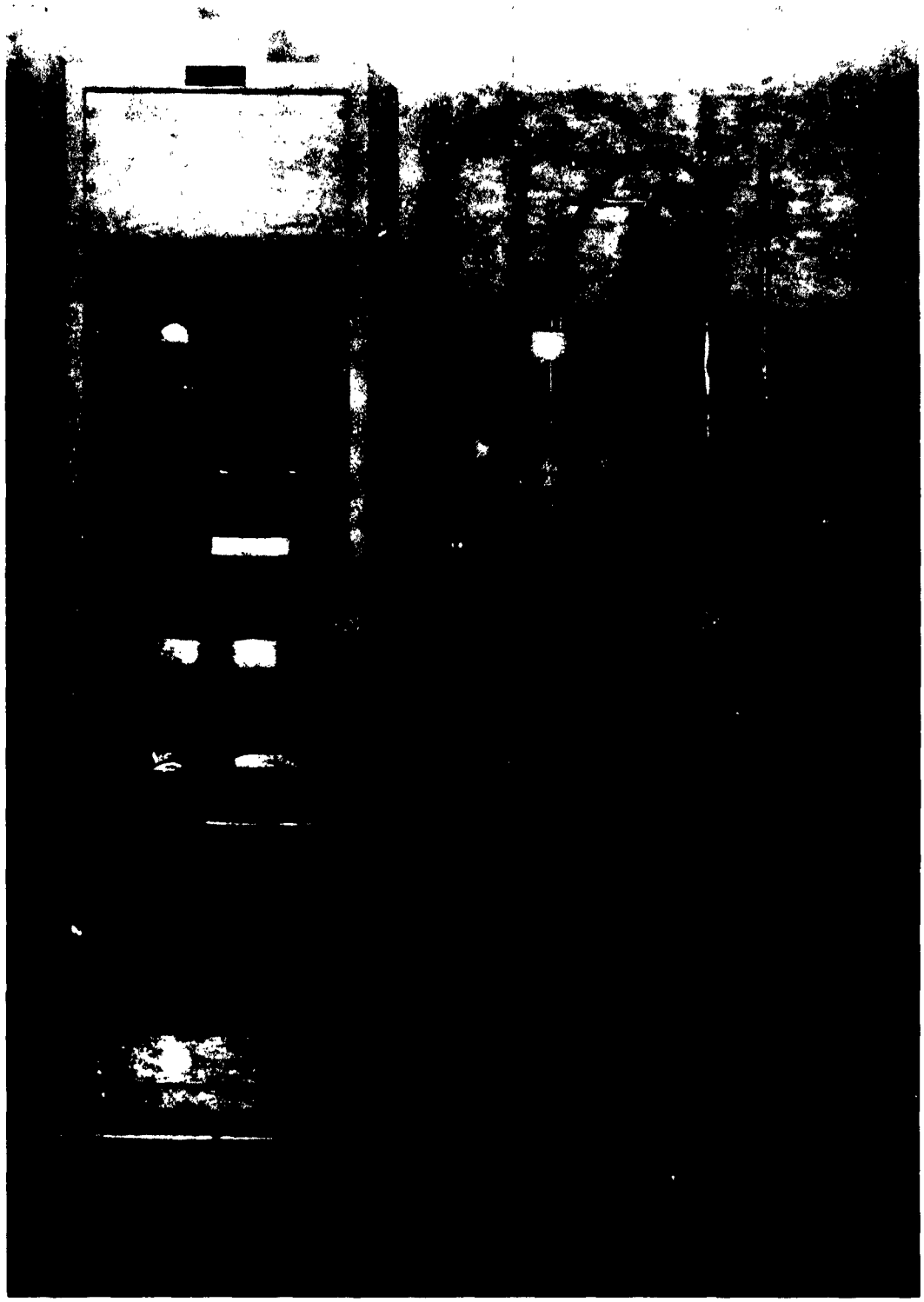

Fig. 3 Field Ion Microscope With Power Supply

liquid nitrogen in the diffusion pump trap, the cold finger, the charcoal trap, and in the microscope head is 3.2×10^{-8} Torr. When liquid hydrogen is put in the microscope head this drops to 1.2×10^{-8} Torr. Typical pump down curves for both a cold start and with an operating diffusion pump are shown in Figure 4. The microscope head is always cooled first to minimuze the condensing of vapors on the tip and cold mantle. The curve shows that if the diffusion pump is already in operation, as will be the case when a specimen change is being made during extended operation, the microscope can be pumped from atmospheric pressure to 6.7×10^{-8} in 31 minutes by liquid nitrogen cooling. If liquid hydrogen were then transferred to the microscope head the microscope would be ready for use in a few minutes. For microscope operation with liquid nitrogen cooling it would be better to wait until the pressure had dropped to below 5×10^{-8} Torr before backfilling with helium.

The 5-3/4 inch vertical screen is much more convenient for visual observation then the usual smaller horizontal screens of the previously used all glass microscopes. A swingaway camera mount allows the camera to be rotated, moved up or down, and sideways parallel to the screen. With the camera swung away the screen may be observed with a magnifier as closely as one chooses. With the shorter tip to screen distance given when the microscope head is in the front opening, the screen is not always filled by

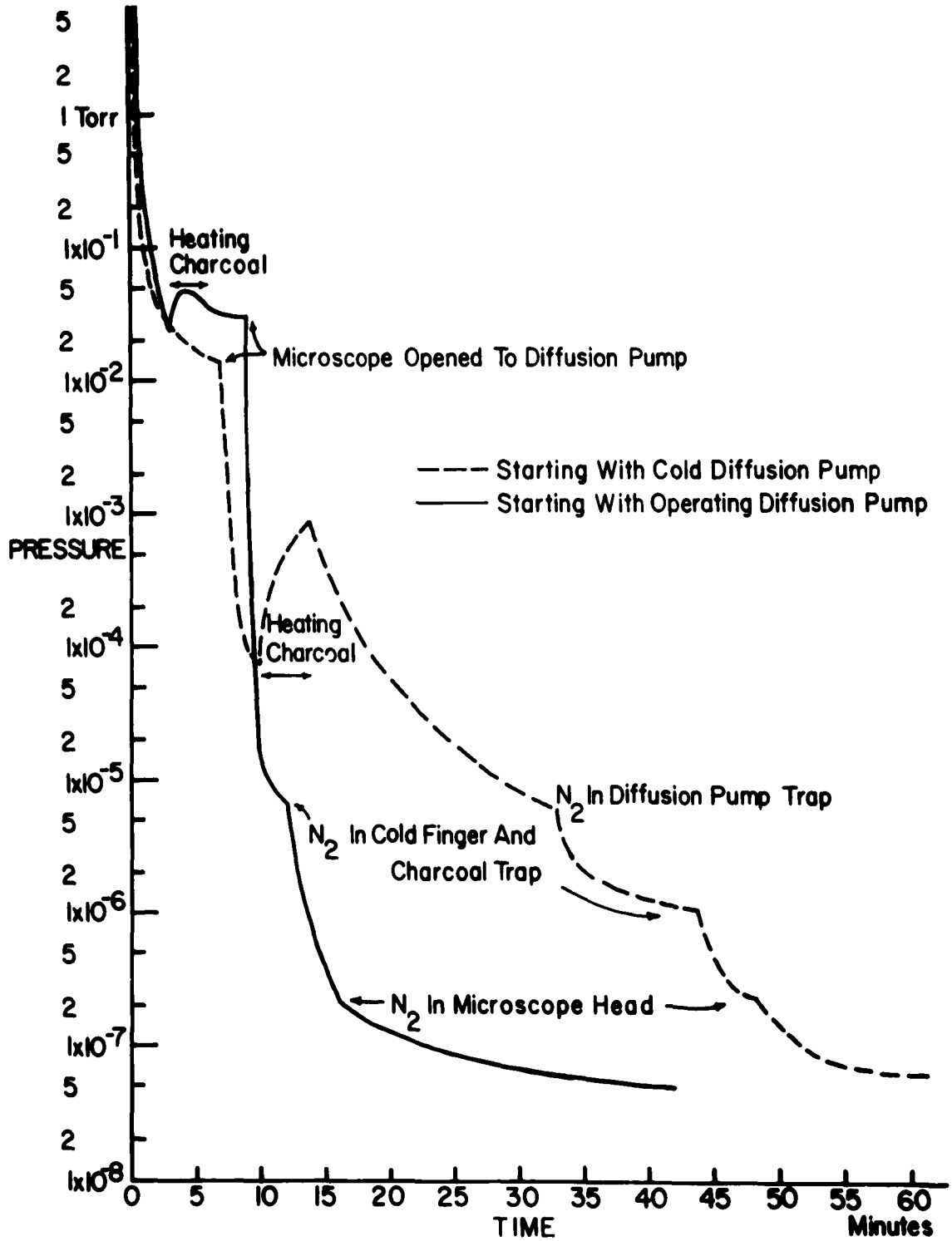

Fig. 4 Typical Pumpdown Curve

the image depending upon the shape of the tip. For greater magnification and therefore less effect of grain in both the screen and film the microscope head can be moved to the rear opening. Comparison photographs for the two different magnifications of the same tip are shown in Figures 6 and 7. With a screen to tip distance of 60.5 mm and a screen diameter of 145 mm the screen would be expected to subtend an angle of approximately 100°; however, due to the well known image angle compression resulting from the geometry of the tip and aperture in the cold mantle it has often been possible to view simultaneously crystal planes whose angles are 180° and more as can be seen in Figure 8.

III. STUDY OF RELATIVE EFFICIENCIES AND RELATIVE LIFE TIMES OF SCREEN MATERIALS

While its versatility enables a wide variety of possible research subjects to be pursued by a microscope of this design, its particular features have been used, to date, for three studies.

First the ease with which fluorescent screens can be applied in this design encouraged a study of relative efficiencies and relative life times of different screen materials, under the bombardment of helium ions.

To accomplish this five screen materials were deposited in sectors on the glass plate. The screen was applied by the puffer method commonly used in this laboratory. This consists of depositing 7 drops of 10% phosphoric acid in

Fig. 5 Insulator and Tip Holder Assembly

Fig. 6 W-Low Magnification 16.6KV 32sec. f/1.4

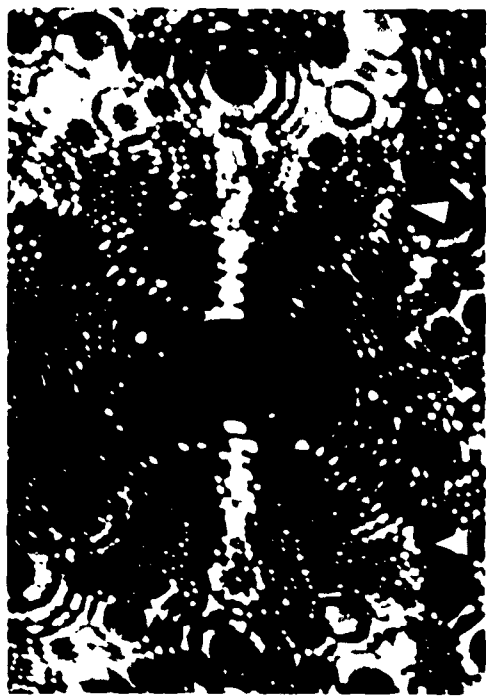

Fig. 7 W-High Magnification 17.1KV 57sec. f/0.87

Fig. 8 W-Wide Angle, 12.4KV 28sec. f/0.87

alcohol solution on the glass plate and spreading uniformly by rolling with glass beads retained to desired area by a metal frame. After the beads were removed, the area was masked and each sector was puffed with the appropriate screen material until the density appeared equal to a standard sample of a density of 0.5 mg/cm^2. Sector A contained the screen material presently in use, Sylvania Lot C140-A $ZnSiO_4$:Mn. Sector B was from a freshly opened bottle of Sylvania Type No. 160, Lot CX-170, $ZnSiO_4$:Mn presumably the same type as that of Sector A. Sector C contained Sylvania Type CR305, Lot CR305-220, ZnS:Ag. Sector D contained Sylvania Lot CR30-70, (ZnCd)S:Ag. Sector E contained Sylvania Type CR407, Lot CR407-103A. The screen plate was not baked after the deposit of the phosphors.

A preliminary experiment showed all five sectors to have almost uniform intensity as viewed by the eye and as recorded by the use of Eastman Spectroscopic Film 103AG. The preliminary experiment ended after a short time due to tip rupture.

Seventeen days later a more extensive experiment was conducted using the same screen and a new tip, made from the same tungsten wire. In this experiment the microscope had first been evacuated to 7.3×10^{-8} Torr and then back-filled through a Vicor diffuser with helium to a pressure of 2×10^{-3} Torr. This pressure was monitored with a thermo-couple gauge and remained constant for the entire

period of the experiment. During the experiment the microscope tip was cooled with liquid nitrogen, and was connected to a charcoal trap also cooled by liquid nitrogen. Photographs were taken at a lens setting of f:0.87.

The tip showed a crude picture at 11 kilovolts and was slowly field desorbed without heating until its best image was at 15KV one hour later. The tip voltage was then raised to 18KV for 44 minutes and later to 19KV. Times referred to in the rest of the report on this experiment indicate time at 16KV or higher. Periodically pictures were taken of sharp images and of blurred images at elevated voltages. The blurred images give a better overall picture of the intensities, and allow for inspection for the effect of burning in by the sharply focused ions. Figure 9 shows a blurred image after 45 minutes at higher voltage. On Eastman 103AG film sectors A,B,C and D responded with virtually equal intensity. Sector E was dimmer to the eye and to 103AG film, however, 25 minutes previous to this exposure the surface brightness in lamberts of the sectors with tip voltage at 19KV as recorded by an Aminco photomultiplier microphotometer were Sector A, 2.26×10^{-7}; Sector B, 1.96×10^{-7}; Sector C, 2.1×10^{-7}; Sector D, 2.15×10^{-8}; and Sector E, 2.45×10^{-7}, the highest.

Since the spectral response of the eye at low intensities is different from that of the photometer and certainly different from that of the 103AG film, which is most

sensitive in the 460-580 millimicron wavelength range, a comparison was made using a panchromatic film, Eastman Tri-X. The Tri-X exposures still showed the Sector E to be dimmer, but the ratio of the intensities of the sectors were closer to unity than with the 103AG film. Effective prints could be made from the Tri-X negatives having the same exposure as the 103AG film, but the Tri-X prints showed less contrast. Both films were developed for 4 minutes at 20°C in Ethol 90 developer. Tri-X film showed such a wide latitude that useable prints could be made from negatives having an exposure range of 16 to one.

The experiment was continued until the same tip and screen had been under high voltage for approximately 27 hours. At this time there was no apparent etch of the tip and the experiment was terminated only because of the failure of the automatic liquid nitrogen level control on the microscope head. Figure 10 shows a blurred image after 26 hours and 26 minutes at voltages above 16KV.

The purpose of this experiment was to determine the practical choice of fluorescent screen material for the FIM. The conclusion reached was that the Sylvania Lot C140-A $ZnSiO_4$:Mn had the longest life time, half life in the order of 8 hours, and also the highest intensity except for Sylvania Type CR407 whose half life was so short in the order 30 minutes that it would be impractical to use.

Fig. 9 Start of Screen Efficiency Test

Fig. 10 Screen Efficiency Test 26hrs. Later

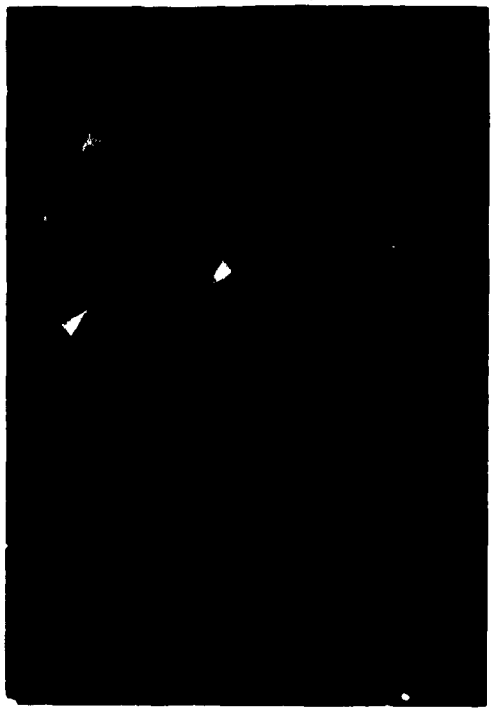

Fig. 11 W-Grain Boundaries 24.7KV 8sec. f/1.4

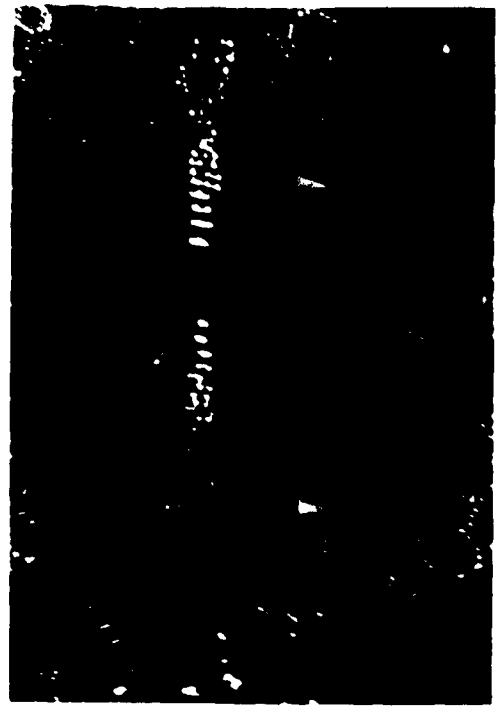

Fig. 12 W-Grain Boundary 17.78KV 22sec. f/1.4

Work on the FIM with dynamic gas supply[5] done under the present contract contains data linking ion current and tip voltage to exposure times or relative image brightness. Since the same screen material (Sylvania Lot C140-A) was used and our present measurements of absolute brightness covered in part the same range we can calculate screen efficiencies under our special operational conditions. As an example we obtain at 16KV with a He pressure of 2 microns an ion current of 1.5×10^{-9} amp. and an image brightness of 1.2×10^{-7} lamberts over an area of approximately 254 cm^2. The screen efficiency for 16KV He ions is then 20,000 Lumen/amp.

IV. THE USE OF SOLID NITROGEN FOR COOLING THE FIM

The satisfactory operation of the field ion microscope depends upon the cooling of the emitting tip. It is well known that a tip cooled to liquid hydrogen temperature $20.4^{\circ}K$ gives an image that is more intense, has better contrast, and better resolution than the same tip cooled to liquid nitrogen temperature, $77.4^{\circ}K$. What is not known precisely is the optimum temperature as a function of the tip radius. Because of the dual disadvantages of liquid hydrogen of the danger of explosion and of higher cost, it was desired to see if satisfactory operation could be obtained by cooling with solid nitrogen or super cooled solid nitrogen obtained by pumping nitrogen. This method had been tried in conventional field ion microscope systems,

but difficulties occurred due to high voltage breakdown through the pumping line. With the modified metal field ion microscope these difficulties are overcome as the Dewar is at ground potential, allowing cooling media to be changed while the high voltage is on and the reduced pressures due to pumping have no electrical effects. A number of trials were made switching the cooling medium from liquid nitrogen to liquid hydrogen and back to liquid nitrogen, from liquid nitrogen to solid nitrogen at 63.3°K and to super cooled solid nitrogen at pressures as low as 12 Torr corresponding to a temperature of 54.5°K. A manometer directly at the microscope head served as the thermometer by conversion from vapor pressure charts. The experimental results were disappointing because of poor heat transfer from the solid coolants to the tip. While the image intensity in this system due to liquid hydrogen cooling was typically twice that of liquid nitrogen cooling, the best increase in intensity due to pumped nitrogen was only 20% higher than that for liquid nitrogen. When the nitrogen was first pumped there was an increase in intensity but as the solid nitrogen sublimed away from contact with the metal transferring the heat from the tip the intensity would again decline approximately to that of liquid nitrogen. Best values were obtained for a solid-liquid mixture, but this was difficult to maintain over long periods. Attempts were made to improve the contact and conduction by introducing a copper sponge but the

violent bumping of the nitrogen as it reached the triple point would knock the stopper out of the microscope head. Liquid neon would be interesting for this study as it has a boiling point of 27.2°K and provides 3-1/3 times more cryogenic refrigeration than an equivalent volume of liquid hydrogen, without any danger of explosion. However the September 1961 price quotation of $250/liter for 1-9 liters and $150/liter over 10 liters still puts its use beyond our present budget.

V. THE USE OF THE FIM TO STUDY GRAIN BOUNDARIES

The field ion microscope is the only known tool for the study of crystal structure in atomic detail. To study defects in crystals we have the inherent problem that the greater detail or magnification with which we use to observe the specimen, the smaller the area which can be observed. With a given defect density, the liklihood of observing a defect will go down with increase in magnification. As the magnification of field ion microscopes is in the order of 10^6 the area covered by the surface of the tip is very small. This particular metal field ion microscope compresses the image as was discussed on page 12. This effect together with the variable magnification possible lends itself well to the study of crystal defects. With low magnification the surface can be searched for defects and if necessary the magnification can be raised to study a particular area if a defect is

found. A study of grain boundaries currently underway has shown that this defect is frequently observed and that with proper preparation of the tip by etching at a point where the metal has been bent that grain boundaries can be observed in higher than 90% of the tips so prepared. Figures 6,7,11 and 12 give examples of grain boundary pictures.

A report on "A study of a Grain Boundary in Three Dimensions" by S. B. McLane and E. W. Müller was presented at the Ninth Field Emission Symposium at the University of Notre Dame in June 1962. A grain boundary in unannealed tungsten was studied in atomic detail by the use of the field ion microscope. The tip with the grain boundary was field evaporated through 368 layers of the 011 plane. A contour of the grain boundary was plotted in a plane through the axis of the tip and determinations were made of the orientation of the plane of the grain boundary with respect to each of the crystals. Details of this study will be included in a later report when more data can be obtained from the study of grain boundaries.

VI. CONCLUSION

Where versatility, ruggedness, and freedom of design are desired, the use of metal in the construction of field ion microscopes has been demonstrated to be both feasible and desirable. The screen of the microscope is the only part where the use of glass is essential.

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